

SCHOOL OF
CIVIL ENGINEERING

INDIANA
DEPARTMENT OF HIGHWAYS

JOINT HIGHWAY RESEARCH PROJECT

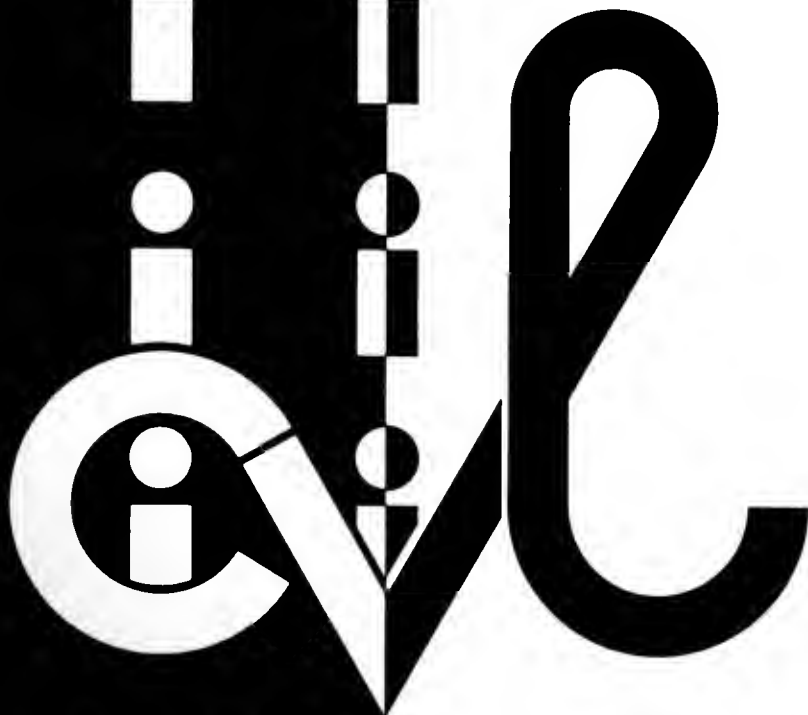
FHWA/IN/JHRP - 86/5

FINAL REPORT

APPLICATION OF A RAPID TEST METHOD
FOR ASPHALT CONTENT

Mary Jo Hamman

Douglas N. Winslow



PURDUE UNIVERSITY



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Final Report

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To: H.L. Michael, Director
Joint Highway Research Project

27 March 1986

From: D.N. Winslow, Research Associate
Joint Highway Research Project

Project: C-36-6HH

File: 2-4-34

The Final Report attached is submitted on the HPR Part II Research Study titled "Application of a Rapid Test Method for Asphalt Content". Mrs. M. J. Hamman has authored the report and performed the research under my direction.

The objective of the study was to extend a recently developed rapid asphalt content test to mixtures for which it was not originally applicable. This has been accomplished, and the test is now applicable to hot emulsion mixes, recycled materials and mixes with a large fines content. These extensions have been accomplished with only modest increases in the complexity of the test, and have added only 1-2 minutes to the required testing time.

The Report is submitted for presentation to the JHRP Board, and for review by DOH and FHWA.

Respectfully submitted,



D.N. Winslow
Research Associate

cc: A.G. Altschaeffl	M.K. Hunter	B.K. Partridge
J.M. Bell	D.E. Hancher	G.T. Satterly
M.E. Cantrall	J.P. Isenbarger	C.F. Scholer
W.F. Chen	J.R. McLaughlin	K.C. Sinha
W.L. Dolch	K.M. Mellinger	J.R. Skinner
R.L. Eskew	R.D. Miles	C.A. Venable
J.D. Fricker	P.L. Owens	E.W. Walters

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Final Report

APPLICATION OF A RAPID TEST METHOD
FOR ASPHALT CONTENT

Mary Jo Hamman
Graduate Research Assistant

Douglas N. Winslow
Associate Professor of Civil Engineering
and
Research Associate

Joint Highway Research Project

Project No.: C-36-6HH

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Conducted by

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Engineering Experiment Station
Purdue University

in cooperation with the

Indiana Department of Highways

and the

U.S. Department of Transportation
Federal Highway Administration

The contents of this report reflect the views of the authors who are responsible for the facts and the accuracy of the data presented herein. The contents do not necessarily reflect the official views or policies of the Federal Highway Administration. This report does not constitute a standard, specification, or regulation.

Purdue University
West Lafayette, Indiana
March 27, 1986

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Highlight Summary

Modifications have been developed for a rapid asphalt content test method that allow it to be applied to 1. mixtures made with hot, emulsified asphalts, 2. recycled (RAP) materials, and 3. mixtures containing a large proportion of fine ,<#200 sieve, material. For mixtures with emulsified asphalts, a modified solvent, greater agitation and a different calibration curve are required. Recycled mixtures require a centrifugation step to clarify the extract and a different calibration curve. Mixtures with large proportions of fines also need a centrifugation step but the original calibration curve will suffice. The method was found to give erroneous results when highly absorptive, small pored coarse aggregates were used.

INTRODUCTION

A recently completed research project (JHRP Report 84-3) developed a rapid test method for determining the asphalt cement content of bituminous paving mixtures. The test method is approximately an order of magnitude faster than many existing methods (2-4 minutes vs. 45-60 minutes). The equipment required is also approximately an order of magnitude less expensive than that required by other methods (\$300 vs. several thousand dollars). The new method is also completely portable and can be used anywhere in the field. All of these advantages have been obtained without any apparent loss in the accuracy of an asphalt content determination. This rapid test method represents a significant advancement in asphalt content determination of bituminous mixtures.

The new test involves placing a standard mass of mixture in a metal can a little larger than a gallon paint can. A standard volume of solvent is then added, the can is closed, and it is shaken briefly. A standard volume of the now asphalt-laden sol-

vent is removed, diluted, and placed in a spectrophotometer, or colorimeter. The light transmittance of this sample is measured, and used to enter a calibration curve. This curve is a plot of transmittance vs. the asphalt content of the original mixture. The calibration curve is based on the testing of mixtures with known asphalt contents. From the curve one obtains the asphalt content of the mixture with the unknown asphalt content.

The original test method was developed for use with hot-mix paving mixtures containing virgin asphalt and a variety of virgin aggregates. This method would be more useful in quality control testing if it were applicable to other mixture types currently in use. The current research project was undertaken to extend the applicability of the new method. The important extensions that were considered in the current study include:

1. Application of the test to hot mixtures made with emulsified asphalt cements.
2. Application of the test to recycled mixtures.
3. Use of the test with mixtures containing a large percentage of fine (<#200 sieve) material.
4. Use of the test with mixtures containing highly absorptive coarse aggregates.

Each of these potential extensions have been studied and the results are given in subsequent sections. The original rapid test method remains as the basis for all the new extensions. The

original test method for hot-mixes is given in the next section. All variations and additions for specific extensions are noted in their respective sections, and referenced to the following section containing the original method.

ORIGINAL ASPHALT CONTENT TEST PROCEDURE
FOR HOT-MIX PAVING MIXTURES

This section covers apparatus, materials and procedures for determining the asphalt content of hot-mix paving mixtures made with virgin asphalt cement and aggregate. Extraction of the asphalt cement is carried out with trichloroethylene. The percentage of transmitted light through the extracted solution is measured and correlated with the asphalt content of the mixture using a standard calibration curve.

I. EQUIPMENT

1. Balance having an accuracy of at least 0.1% of the sample mass.
2. Hach colorimeter, Model DR-100, (610 nm wavelength) with good quality cells that can be read to 1% of transmittance of light.
3. Aluminum extraction container.

4. Pipet, 1 ml, with pipet filling bulb. A reliable dispensing pipet may be substituted for this item.
5. Volumetric flask, 2000 ml.
6. Volumetric flask, 100 ml.

II. REAGENTS

Trichloroethylene, technical grade. NOTE: This solvent is toxic and should be used only under a hood or in a well ventilated area. The maximum acceptable concentration of vapor for an 8 hour exposure is reported to be 100 ppm.

III. PROCEDURE

1. A representative sample of the mixture (approx. 8 kg) shall be placed in a large, flat metal pan. If the mixture is not sufficiently soft to separate with a spatula, it should be warmed in an oven at approximately 230 F (110 C) until it can be handled.
2. Divide the sample into four approximately equal parts. Weigh 2 kg into the extraction container.
3. Add 2000 ml of trichloroethylene to the extraction container, place the rubber O-ring in the groove, and place the lid on the container. Put the metal ring around the extraction container and secure the clamp.

4. Close the bleed valve before starting the extraction procedure.
5. Hold the container, by the metal ring, with both hands, and invert it. Agitate it with a twisting movement for several seconds. Then bring it back to the normal, "cover up" position. Repeat this operation eight times without a pause.
6. Place the extraction container under a hood (if testing indoors) and open the bleed valve to release the pressure. Close the bleed valve.
7. Repeat the agitation process of Step No. 5 seven more times.
8. Again, place the extraction container under a hood (if testing indoors) and open the bleed valve to release the pressure.
9. Remove the metal ring and the lid.
10. Add 100 ml of trichloroethylene to the 100 ml flask. Take a sample of the extracted asphalt solution with the 1 ml pipet approximately one inch below the surface of the liquid in the extraction can and add it to the 100 ml flask.
11. Rinse the 1 ml pipet with the solution in the 100 ml flask and return the rinse to the flask.

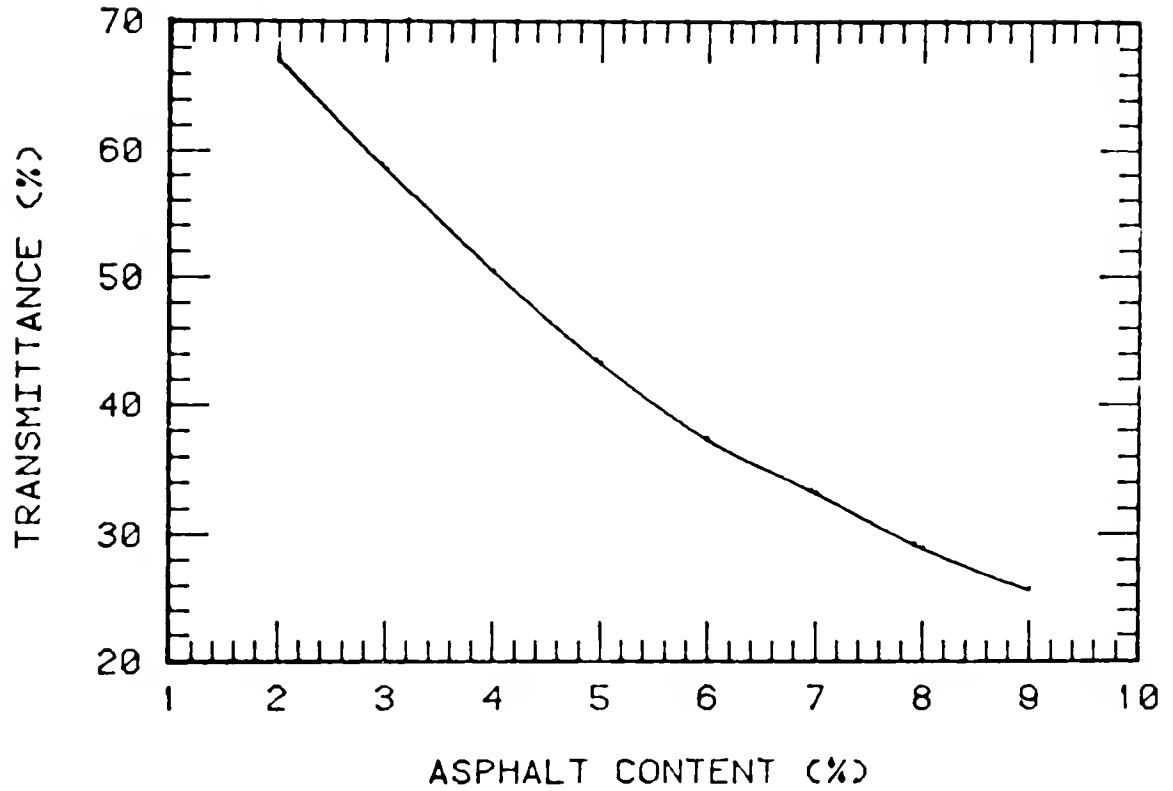
12. Cover the flask and mix by shaking ten times.
13. Transfer some of the diluted solution to a colorimeter cell, and rinse it twice with the solution. Then fill it with the solution that is to be tested.
14. Fill another cell with pure trichloroethylene to be used as a blank.
15. Adjust the colorimeter to 0% transmission with the light path blocked and to 100% transmission with the pure solvent. Repeat the adjusting procedure at least twice.
16. Place the cell with the sample solution in the colorimeter and measure the transmittance of light.

IV. CALCULATION

Find the percentage of asphalt in the original mixture by entering the calibration curve, Figure 1, with the measured transmittance and reading the corresponding asphalt content from the other axis.

Figure 1

Calibration Curve for Original Test Procedure



MODIFICATION OF THE TEST FOR
MIXTURES MADE WITH HOT EMULSIFIED ASPHALTS

The original research determined that pure tri-chloroethylene, (TCE), would not completely dissolve an emulsified asphalt cement. The asphalt emulsions used were partially immiscible in pure TCE. Apparently the emulsifying agent was responsible for the difficulty.

Tests were carried out in the current study using a series of modified solvents to find a solvent that would dissolve the emulsions. Combinations of TCE with ethyl alcohol (ethanol) and TCE with methyl alcohol (methanol) in varying proportions were examined. The most effective solvent mixtures were found to be those that contained about 10 volume percent alcohol. Both alcohols appeared equally effective. Methanol was chosen because it is free of the tax problems associated with purchasing ethanol. A modified solvent containing 10% methanol and 90% TCE, BY VOLUME, was used in all subsequent emulsion testing.

Three different emulsified asphalt cements were examined in order to verify that the solvent was reasonably general in its applicability. All of the emulsions were Type AE-60. They were manufactured by the following companies.

Manufacturer	Location	Emulsion Code
McConnaughay Mixers	Lafayette, Indiana	MM
Asphalt Materials	Indianapolis, Indiana	AM
Ashland Oil	North Vernon, Indiana	AO

It was necessary to determine which calibration curve, the existing one or a new one, was useful with this new solvent. This was done, as in the original research, by testing mixtures of known asphalt content. Three gradations of particular interest in Indiana were tested using gravel as the coarse aggregate. These gradations have the Indiana Department of Highways surface mix designations HAE No. 9 Surface, HAE No. 11 Surface and HAE Sand mix. Their gradations are given in Table 1.

Table 1

Gradations of Aggregate for Emulsion Testing

Sieve Size	No. 9 (% retained)	No. 11 (% retained)	Sand (% retained)
3/4"	0	0	0
1/2"	12	0	0
3/8"	30	7	0
# 8	52	46	13
# 16	62	59	33
# 30	79	76	63
# 50	88	88	80
# 100	95	95	91
# 200	98	98	98

Each mix gradation was prepared with the variety of asphalt contents given in Table 2. It was found that the modified solvent produced good results if the agitation of the extraction chamber was increased. The details of these modifications are given in the following section.

The results of applying the modified method to the series of mixes with known emulsified asphalt contents are given in Table 2.

Table 2

Calibration Results for Mixes with Hot Emulsified Asphalts

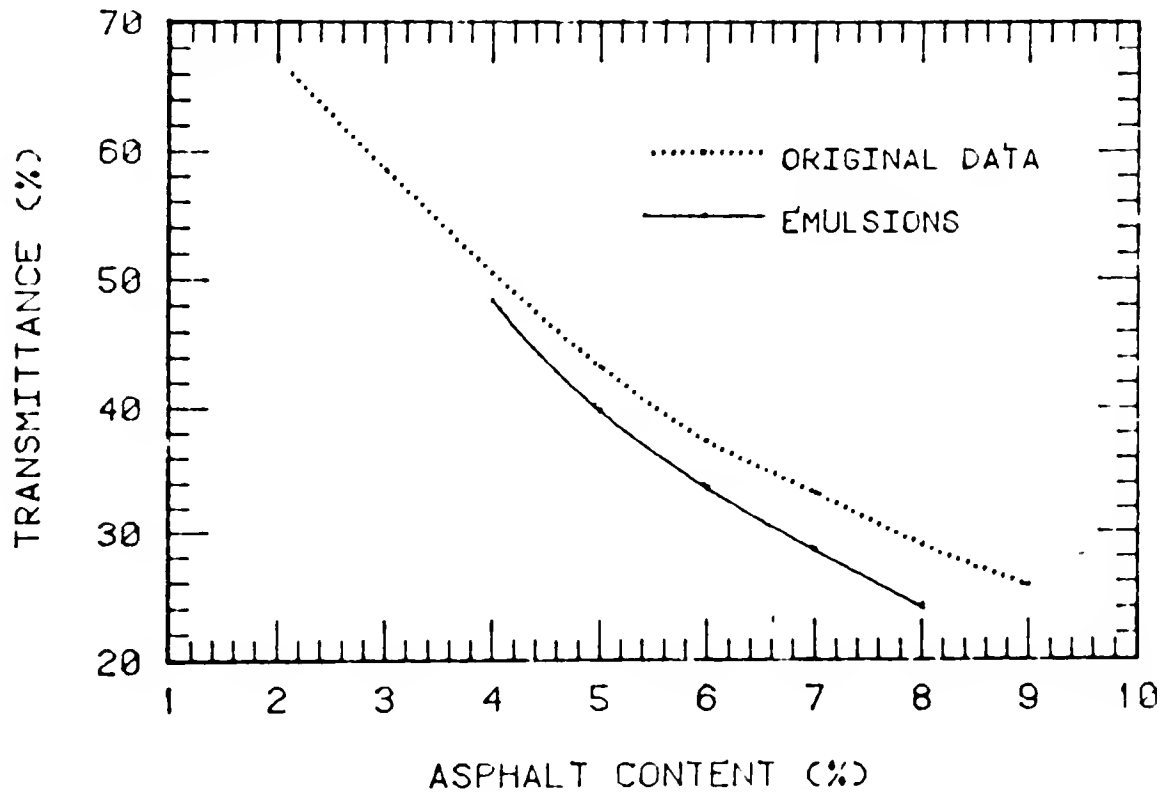
Mixture Type	Asphalt Content (%)	Transmittance (%) for Different Emulsions		
		MM	AM	AO
No. 9	4	48	48	49
	5	38	40	41
	6	31	34	34
No. 11	5	37	42	40
	6	32	34	34
	7	29	29	28
Sand	6		36	33
	7	28	29	28
	8	21	24	23

The individual test results do not appear to vary greatly, between mix types or emulsion manufacturers for the same asphalt content. Therefore, the average transmittance for all results at the same asphalt content was used to develop a calibration curve. This curve is shown in Figure 2. For purposes of comparison, the calibration curve developed in the original research program, for

plain asphalt cements, is also shown in Figure 2. The details of the modifications to the test are given in the next section.

Figure 2

Transmittance of Emulsion Extracts vs. Asphalt Content
(Hot-Mix Calibration Curve is Shown as a Dotted Line)



Asphalt Content Test Procedure for
Hot Emulsified Asphalt Cement Mixtures

I. Equipment

All equipment required is the same as that required for the hot-mix paving materials test.

II. Reagents

Trichloroethylene and methanol, technical grade. NOTE: trichloroethylene is toxic and should be used only under a hood or in a well ventilated area. The maximum acceptable concentration of vapor for an 8 hour exposure is reported to be 100 ppm.

III. Procedure

Changes in the procedure from that of the original test are given below. All steps that are not specifically mentioned remain the same as those in the original procedure.

- a. Step 3. Add 2000 ml of the solvent (90% TCE, 10% methanol, by volume) to the extraction container, place the rubber O-ring in the groove, and place the lid on the container. Put the metal ring around the extraction container and secure the clamp.
- b. Between steps 8 and 9, add step 8A: Repeat the shaking process 15 more times as per Steps 5, 6 and 7. This

additional step is necessary to insure that all the emulsifying agent is dissolved by the modified solvent.

- c. Step 12. Cover the flask and mix it by shaking 20 times. Again, this insures that all the emulsion is dissolved.

IV. Calculation

Find the percentage of asphalt in the original mixture by entering the calibration curve for the emulsion mixes, Figure 2, with the measured transmittance and reading the corresponding asphalt content.

Discussion of Emulsion Mixture Testing

The rapid test method seems to be completely suitable for testing bituminous mixtures made with hot, emulsified asphalt binders. It is only necessary to use a modified solvent and to increase the amount of agitation. However, Figure 2 clearly indicates that a different calibration curve is required. This is probably the result of using a different solvent or of the presence of the emulsifying agent in the extract, or both.

The three emulsions used in this study did not yield significantly different transmittances at the same asphalt content. Thus, the single calibration curve is applicable to mixtures made

with any of them. However, other emulsions might require a different calibration curve. The need for a different curve can be quickly checked with several mixtures of known asphalt content. And, a new curve can be quickly established since the emulsion extracts, like those from the hot-mixes in the original research, follow Beer's Law, and a plot of absorbance vs. asphalt content is a straight line.

MODIFICATION OF THE TEST FOR RECYCLED MIXTURES

Recycled mixtures contain old, weathered asphalt, foreign material that a roadway has picked up in service, and possibly an increased quantity of fines due to grinding of the aggregate by traffic and by the pavement removal process. Any of these has the potential to interfere with either the extraction process or the light transmittance, or both. The original research determined that the standard hot-mix rapid test produced a lower transmittance of light and an erroneously high asphalt content when applied to recycled material. This was due to dark, suspended particles in the diluted extract that would not settle to the bottom of the colorimeter cell rapidly. One goal of the current research was to eliminate the interference caused by these suspended particles, and to make the rapid test applicable to recycled mixes.

One procedure that was considered was filtration of the extract with a non-absorbing, polymetric filter. Teflon filters were used. However, the chemical coatings on some of these

filters interfered with the solvent solution and lead to erroneous measurements of the light transmittance. In other cases, with uncoated filters, the act of passing the extract through the filter appeared to break the dark suspended matter into smaller pieces that made the situation worse. For these reasons, filtering was found to be an unacceptable process.

A second option was some type of centrifuging process. Since it was found that the dark particles would settle on their own in several hours, the effect of a centrifuge would be to speed this settling. Initially, a hand held centrifuge was tried. The variability of this apparatus and the relatively long centrifuging time, approximately 5 minutes, made this simple solution a poor choice.

However, a simple and cheap electrically operated centrifuge was found to produce a clear extract in seconds. Using such a centrifuge adds the complication that electricity must be available at the testing location, but it does permit testing of recycled mixes. The centrifuge that was used operated at about 1600 rpm and had a swing radius of about 3 inches. It is believed that any similar, simple centrifuge would produce equally good results. It was found that 30 seconds of spinning was sufficient to raise the transmittance of the extract to its maximum value. Additional spinning did not clear the extract further.

The next step was to find the calibration curve that was applicable for mixtures from which the extract had been centri-

fuged. This was attempted by testing samples of recycled asphalt pavement (RAP) from a variety of highways. The samples were first subjected to the rapid test, with centrifuging, and the transmittance of the cleared extract was measured. Next, the entire contents of the extraction chamber was tested for asphalt content using one of the variants of the standard test method for asphalt content (ASTM D-2172, Method B).

A total of 30 RAP samples were tested in this manner. They ranged in asphalt content from 4.1% to 7.2%, and came from a variety of pavements throughout Indiana. The results of the individual tests are shown in Table 3. They were all obtained using the modified centrifuge method that is described in detail in the next section.

Table 3

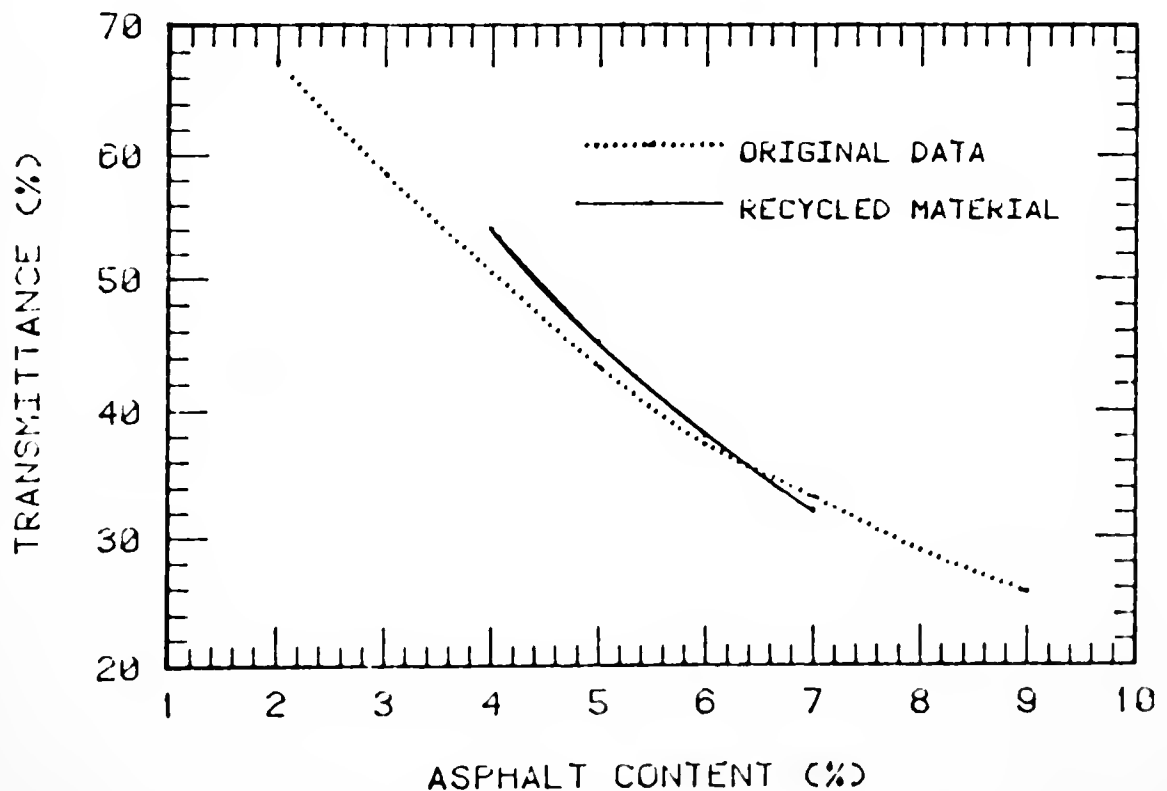
Asphalt Contents and Transmittances of RAP Samples

Asphalt Content (%)	Transmittance (%)	Asphalt Content (%)	Transmittance (%)
4.1	47	5.6	41
4.2	49	5.6	45
4.3	42	5.6	45
4.6	49	5.7	42
4.6	44	5.7	46
4.7	44	6.1	35
4.8	44	6.4	35
4.9	44	6.6	43
5.0	46	6.8	41
5.2	38	6.9	35
5.2	43	7.0	34
5.4	38	7.1	33
5.4	42	7.1	34
5.4	37	7.2	33
5.6	41	7.2	35

The combined data from Table 3 are shown in Figure 3. Note that the transmittances are plotted against the measured asphalt content as per ASTM D-2172 (B), and not against the true asphalt content as has been the case in Figures 1 and 2. For comparison, the original, hot-mix calibration curve is also shown in Figure 3. The details of the modifications to the test are given in the next section.

Figure 3

Transmittance of RAP Extracts vs Measured Asphalt Content
(Hot-Mix Calibration Curve is Shown as a Dotted Line)



Rapid Asphalt Content Test Method Procedure
For Recycled Asphalt Cement Mixtures

I. Equipment

All of the equipment required for the hot-mix paving materials test is required in this procedure also. Additionally, a simple centrifuge with about a 3" swing radius and operating at about 1600 rpm is needed.

II. Reagents

Trichloroethylene, technical grade. NOTE: This solvent is toxic and should be used only under a hood or in a well ventilated area. The maximum acceptable concentration of vapor for an 8 hour exposure is reported to be 100 ppm.

III. Procedure

Changes in the procedure from that of the original test are given below. All steps that are not specifically mentioned remain the same as those in the original procedure.

- a. Between steps 12 and 13, add step 12A: Transfer some of the diluted solution to two centrifuge tubes and place them on opposite sides of the centrifuge to balance it. Run the centrifuge for 30 seconds. This step is necessary to promote sedimentation of the suspended black particles.

- b. Step 13: Once the centrifuge has come to a complete stop, transfer some of the diluted, centrifuged solution to a colorimeter cell being careful not to disturb the sedimentation. Rinse the colorimeter cell twice with the solution, then fill it with the solution to be tested.

IV. Calculation

Find the percentage of asphalt in the original mixture by entering the calibration curve for recycled mixes, Figure 3, with the measured transmittance and reading the corresponding asphalt content from the other axis.

Discussion of Testing Recycled Bituminous Mixtures

The data in Table 3 show some scatter. This is probably to be expected because there was no way to determine the true asphalt content of the samples. Thus, any error in either the rapid test or in the ASTM test would contribute to scatter in the results.

One way to compare the scatter of the RAP results to that in the original study is as follows. The absorbance of a solution is defined as the logarithm of the reciprocal of the transmittance. Further, Beer's law states that a plot of absorbance vs. concentration (asphalt content) should be a straight line. If

one uses linear regression on a set of absorbance and concentration data, the scatter in the data would be reflected in the correlation coefficient of the resulting line that best fits the data. Table 4 lists the correlation coefficients of such plots for the original study data and the RAP data over the same range of asphalt contents.

Table 4

Correlation Coefficients of Absorbance vs Asphalt Content Plots
(asphalt contents ranging from 4-7%)

Material	Correlation Coefficient
Original Study, Virgin Material	0.912
Recycled Material	0.911

It is evident from Table 4 that, in spite of the uncertainty of the asphalt content, the data for the RAP samples have no more scatter in them than do the original data.

The calibration curve shown in Figure 3 for recycled mixtures was developed in the same way as that in the original study. The transmittances were converted to absorbances and a Beer's law plot was made. A best straight line was found for the data, and this line was used to generate transmittance values for selected asphalt contents. It appears that the weathering of the asphalt has changed its optical properties and that, while close to the original curve, a different calibration curve is required for recycled mixtures.

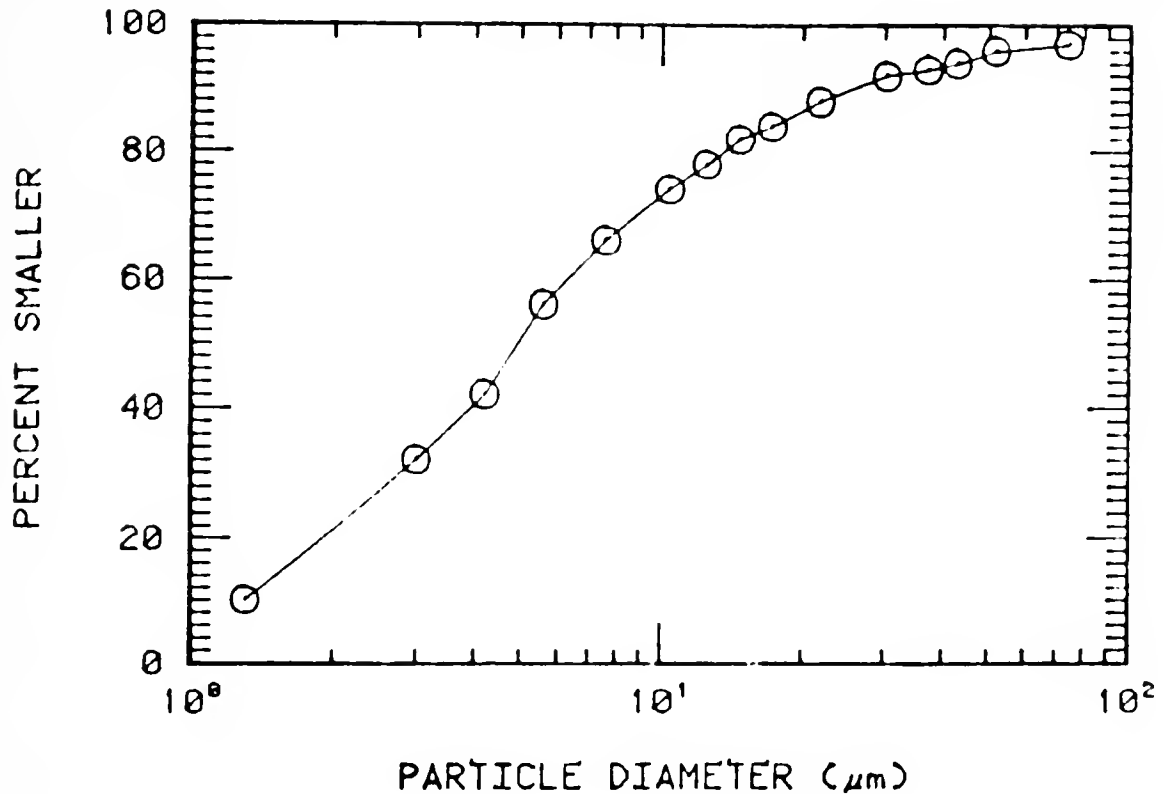
MODIFICATION OF THE TEST FOR FINE MATERIALS IN MIXTURES

The original research was done on mixtures that contained a maximum of about 4% fines (aggregate < #200 sieve). These fines did not appear to interfere with the test method. However, further experiments showed that if these fines were extremely well dispersed in the solvent, they would interfere with the test. It is probably their normal lack of complete dispersion that keeps them from interfering in testing a normal mixture. However, mixtures are sometimes made with considerably larger quantities of fines such as collections from bag houses. When these mixtures were tested with the original procedure, they did interfere with the light transmittance of the extract, and gave erroneous results.

A sample of bag house fines was obtained from the Indiana Department of Highways and used to test bituminous mixtures with a relatively large percentage of this fine material. The particle size distribution of these fines, as determined by sedimentation, is shown in Figure 4. A standard #11 mixture was modified

Figure 4

Particle Size Distribution of Bag House Fines



to contain 10% fines (passing a #200 sieve) by proportionally reducing the amounts of the larger aggregate sizes. This mixture was tested using the procedure for recycled mixtures since the problem created by the dispersed particles is essentially the same as that encountered with the RAP samples. The results of several trials on mixtures containing 4% asphalt are shown in Table 5.

Table 5

Tests on Mixtures Containing 10% Fines

Trial No.	Transmittance w/o Centrifuging	Transmittance w/ Centrifuging
1	20%	49%
2	20%	50%
3	20%	49%

Discussion of Testing Mixtures

with a Large Proportion of Fine Materials

The expected transmittance of mixtures containing 4% asphalt from the original study, see Figure 1, is 50. The data in Table 5 indicate that, although the fines greatly reduce the transmittance when they are in the light path, centrifuging the extract removes them and makes the rapid test applicable to such mixtures. Thus, the same modifications that make the test useful for recycled mixtures appear to be valid when testing mixtures with a large fines content.

TESTING MIXTURES WITH HIGHLY ABSORPTIVE AGGREGATES

In the earlier study, a few mixtures were tested that contained slag as the coarse aggregate. Slags are, typically, highly porous, and would be expected to more strongly retain asphalt. It was found that a modest increase in the agitation of the extraction can would promote the release of the absorbed asphalt cement. However, some aggregates, with reasonably large pore volumes, have much smaller pore sizes than slags, and are sometimes used in mixtures. Asphalt enters these pores, and some of it is not removed by the existing, standard extraction procedures. The last task of the current study was to examine the applicability of the rapid test method to bituminous mixtures made with these highly absorptive aggregates.

A sample of a highly absorptive aggregate was obtained from the Indiana Department of Highways. This aggregate had a 24-hour absorption of about 6%. All of the previous mineral aggregates that had been used had absorptions of 1 - 2%. Standard #11 mixtures were prepared using the absorptive aggregate for the coarse

aggregate. The results of several trials on mixtures containing 4% asphalt are given in Table 6.

Table 6
Tests with Highly Absorptive Coarse Aggregates

Trail No.	Transmittance
1	60 %
2	59 %

Discussion of Testing Mixtures with
Highly Absorptive Coarse Aggregates

The expected transmittance for the mixtures given in Table 6 is 50%. Table 6 shows that the transmittances are much higher than they should be. Indeed, the asphalt content of a mixture would need to be less than 3% to get this high a transmittance. This indicates that some of the asphalt is not being removed from the pores of the highly absorptive aggregates. Further, when the amount of agitation was increased to 10 minutes, the transmittance still did not fall. Thus, it is concluded that the rapid test cannot be expanded to mixtures with aggregates that have a high absorption.

CONCLUSIONS

1. The rapid test method can be expanded to include hot, emulsion mixtures by using a different solvent and increased agitation. A different calibration curve is required.
2. The test can also be used to test recycled material if the extract is first clarified with a brief centrifugation. A different calibration curve is required.
3. The use of a centrifuge will also permit the test to be used on mixtures containing large amounts of fine (< #200 sieve) aggregate. The original calibration curve can be used.
4. The new test gives erroneously low asphalt contents when a mixture contains an aggregate with a high absorption.

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